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DAICEL CHEM IND LTD

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91.04.04 91JP-071882 (92.10.29) C08G 65/20 Prepn. of polysoxystetra:methylene:glycal efficiently by polymerising tetra:hydro:furan in presence of solid catalysts of composite metal oxide and carboxylic acid anhydride, used for e.g. engineering plastic Č92-182149

Prepn. of polyoxytetramethyleneglycol comprises polymerising tetrahydrohuran in presence of both:

 $M_{x}O_{y}$

(a) solid catalysts composed of composite metal oxides of the formula (I), and

(b) carboxylic acid anhydrides.

M = metal; and x and y = 1,2 or 3.

ADVANTAGES/USES

The solid catalyst is easily recovered from the reaction mixt. and reactivated. The polyoxytetramethyleneglycol (POTG) is obtd. easily and is used as a raw material for polyurethanes, polyetheresters, polyetherA(2-A6, 2-A7, 5-H5, 10-D3)

(ester)amides, surfactants, or engineering plastics or for medical uses.

The catalysts pref. comprise metal oxides such as Al₂O₃, SiO₂, TiO₂, ZrO₂, WO₃ or ZnO₂. Pref. catelysts include Al₂O₃-SiO₂, SiO₂-TiO₂, SiO₂-ZrO₂ and TiO₂-ZrO₂.

The catalyst is prepd. as follows; (i) metal conig. -cpds. e.g. metal alkoxides, metal chlorides, metal oxychlorides are added to ammonia to ppte. composite metal oxides, and (2) the pptes. are crushed to particles (200-500 mesh pass) and calcined at 300-600°C.

Silica-alumina solid catalyst (alumina content = 70%) obtd. by calcining at 500°C for 4 hrs. in an air atmos. is packed in a piston-flow type reactor (dia. = 30mm; length = 200mm).

20ml/hr. of THF contg. 3.8 wt. % of acetic anhydride is fed at 40°C and polymsn. is carried out for 132 his. continuously to give 404.3g of polyoxytetramethyleneglycol diacetate ($\overline{M}n = 1,020$).

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The catalyst is removed from the reactor and washed with THF. After drying, the catalyst is charged to a tube (dia. = 30mm, length = 400mm) and calcined at 500°C for 7 hrs. under air.

THF is polymerised by the same way in presence of the reactivated catalyst to give 406.2g of polyoxytetramethyleneglycol diacetate (Mn = 1,060). (5ppW156DwgNo0/0).

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